

**AFRL-ML-WP-TP-2006-502**

**CORROSION RATE EVALUATION  
PROCEDURE (CREP): A  
CONVENIENT RELIABLE METHOD  
FOR DETERMINING CORROSION  
INHIBITION ABILITY OF  
LUBRICANTS (PREPRINT)**



**Marcie B. Roberts, George Fultz, Lois J. Gschwender, and Carl E. Snyder**

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# Corrosion Rate Evaluation Procedure (CREP): A Convenient Reliable Method for Determining Corrosion Inhibition Ability of Lubricants

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## Abstract

The Corrosion Rate Evaluation Procedure (CREP) was first designed as a screening test for corrosion inhibited aerospace gas turbine engine oils as a substitute for the Humidity Cabinet Corrosion Test (ASTM D1748) to screen large numbers of oils in a short period of time for their ability to prevent rust. The CREP was later adapted for hydraulic fluids and greases because of its effectiveness, simplicity, low cost, and speed. For greases it is an alternate method to ASTM D1743 and can evaluate corrosion protection in 1.5 to 2 hours using only 2-4 milligrams of grease. The metal coupons, used in the CREP test, are low cost and available in a variety of metallurgies. The CREP is effective, convenient, reliable, adaptable, cost effective, and usable with greases, liquid lubricants, or hydraulic fluids and various metals as desired.

## Background

This paper describes chronologically how CREP evolved as new lubricant development programs required a fast and reliable corrosion screening technique. The Appendix contains a general procedure that may easily be used and modified as needed.

## Corrosion Inhibited Engine Oils

The CREP was first developed to screen corrosion inhibited gas turbine engine oil candidates at Pratt & Whitney Aircraft Group in conjunction with the Air Force Materials Laboratory<sup>1</sup>. For CREP several test parameters including surface preparation, airflow rate, vapor temperature, and test length, were varied to find the most repeatable and discriminating test method. CREP correlated well with the then-used 144 hour humidity cabinet corrosion (HCC) test, ASTM D1748<sup>2</sup>. For the HCC test panels are prepared using sandblasting or sanding, cleaned and stored in a desiccator. The panels are dipped several times in the oil to be tested and hung to drain for about 2 hours prior to suspension in the humidity cabinet. The air temperature, pH of water, air rate, and water level in the humidity cabinet must be checked twice each day except on weekends. For an hours-to-failure test the panels are checked once each day and placed back into the

humidity cabinet. Otherwise panels are placed in the cabinet and removed after a specified period of time, cleaned, and inspected. For MIL-PRF-8188<sup>3</sup> the test is run for 144 hours and no more than 1 in 5 panels may fail for the lubricant to pass. Failure occurs when there are any dots of rust larger than 1mm in diameter or more than 3 dots of any size. Three or fewer dots, all smaller than 1 mm in diameter, are passing. This test is only for use with liquid lubricants. The following CREP method was developed.

### CREP Sample Preparation<sup>4</sup>

Boil two AISI 1010 steel coupons measuring 5.08 x 1.27 x 0.16 cm (2 in x 0.5 in x 0.06 in) in acetone and toluene for 5 minutes each, and then cooled to room temperature for 30 minutes in a desiccator. Vapor blast the coupons with a slurry comprised of 30 to 40 percent by volume 200-grit novaculite in water on all sides. Boil in acetone and toluene for 5 minutes, and then cool for 30 minutes in a desiccator. Dip the coupons into lubricant then hang in a dust free environment for 15 minutes. Remove the excess fluid from the bottom of the coupon by dabbing it with a lab wiper or paper towel.

### Test Apparatus Assembly

After carefully cleaning and drying all glassware, place the 1-liter reaction kettle on the hot plate. Add approximately 50 boiling beads to the reaction kettle. Set up glassware (condenser, reaction kettle and lid, splash suppressor, standard taper 34/45 to 24/40 reducing type bushing adapter, threaded offset adapter, glass air inlet tube, glass stoppers modified with glass hooks) according to Fig. 1. Support the condenser as required.

Form the coupon suspension hooks from AMS 5680 stainless steel 20 gauge wire so that the bottom edge of the test coupon is 6 cm from the Teflon separator near the bottom of the kettle. The exact measurements changed later with different grease kettle dimensions.

### Test Procedure

Start the water flow through the condenser. Add 100 ml of acetic acid/sodium acetate buffer (Fisher Scientific pH 4.63 Certified Buffer Solution) to the reaction kettle. Establish an airflow rate of 500 milliliters per minute to the reaction kettle before the water starts to boil. Turn on the hotplate such that the reflux line occurs approximately halfway up the metal coupon. The temperature may be adjusted as needed to achieve this. Replace one of the glass stoppers with a thermometer in a 24/40 stopper to determine when the temperature has equilibrated. Allow the system to equilibrate for 60 minutes before test specimens are introduced. Suspend the fluid coated test coupons by the coupon suspension hooks from the glass stoppers and insert in the reaction kettle. Position the coupons radially from the center of the kettle. See Fig 2. Remove the test coupons from the reaction kettle after 60 minutes.

### Results

Remove remaining oil and loose material from the coupons by wiping gently with a wire brush. Clean coupons by wiping all surfaces with a microwiper saturated with acetone and one saturated with toluene until no more loose material can be removed.

Immerse each coupon in boiling acetone followed by boiling toluene. Flash dry and place in a desiccator to cool.

Weigh each coupon.

$$\text{Corrosion Rate} = (\text{weight}_{\text{initial}} - \text{weight}_{\text{final}}) / \text{test length}.$$

A lower corrosion rate correlates to better rust inhibition by the oil.

## **Nonflammable Aircraft Hydraulic Fluid Development**

As new material development programs began, the CREP was modified to take advantage of its simplicity, low cost, and speed to evaluate many materials<sup>5</sup>.

The CREP test was used in the Nonflammable Aircraft Hydraulic Fluid Development Program to screen numerous chlorotrifluoroethylene oligomer (CTFE) formulations with additives. The procedure was followed as it had been developed for the Corrosion Inhibited Engine Oils with several modifications: Of the three coupons one was dipped in test oil while the other two were dipped in standards, one being a corrosion prone standard and the other a corrosion resistant standard.

### **Sample Preparation**

Three coupons were prepared by hand polishing with 240 and then 320 grit silicon carbide papers rather than vapor blasting. (In comparing vapor blasted vs. hand sanded coupons hand sanding gave similar results and did not require vapor-blasting equipment.) Metals were not preweighed because all attempts to use weight change as a criterion for rust protection were unsuccessful, due to the non-reproducible nature of the corrosion product on the metal specimens.

### **Procedure**

Deionized water was used in place of buffer because buffer was too aggressive for this type of lubricant. The 1-liter reaction kettle was replaced with a 2-liter reaction kettle to allow better vapor circulation.

### **Results**

Remove the coupons from the reaction kettle and wipe lightly with a lab wipe as had previously been done. Photograph the coupons on each side. Rate the coupons numerically from 1 to 10, as described in the following paragraph, rather than weighing the coupons before and after the test and calculating corrosion rate. This change was made because the coupons gain and lose weight randomly during this test. The weight loss of the coupons was not a consistent measure of the corrosion of the coupon. The non-repeatability of the weight change became a big problem especially with different people and different laboratories conducting the test. Also in comparing results from different laboratories, it was found that the surface of the metal coupons could be over polished and become less prone to corrosion. For this reason, the time polishing must be limited to 30 seconds per side per sand paper grit.

Numerically rate the appearance of the coupon of the tested fluid, comparing it to the coupons that were immersed in the standard reference fluids. The coupons corresponding to standard reference fluid A (with corrosion inhibitor) and standard reference fluid B (base stock) are rated 10 and 1 respectively. Visual ratings are based on a linear interpolation of the sample coupon compared to the two reference coupons. A visual

rating of less than 8 is typically considered a failure. If the two reference standards are not corroded properly, essentially uncorroded for reference fluid A and severely corroded for reference fluid B, the test results are considered invalid.

The use of this modified CREP was instrumental in the successful development of a corrosion inhibited nonflammable hydraulic fluid.

## **Perfluoropolyalkylether (PFPAE) Oils for High Temperature Gas Turbine Oil**

The CREP was used in the program to screen additives for use with linear PFPAE fluids with  $\text{OC}_3\text{F}_6$  repeating units<sup>6</sup>. As with CTFE, deionized water was used because the buffer solution was too aggressive. The tests were run with two reference coupons: one bad reference coupon having been dipped in the unformulated PFPAE and the second good reference coupon having been dipped in the PFPAE formulated with 1% of a known antirust additive. A third coupon was dipped in the test formulation. One modification was made to the CREP as used in the nonflammable aircraft hydraulic systems development program: The tests were run for 16 hours to get adequate corrosion to see a difference. The coupons were rated based on the criteria previously described in the nonflammable aircraft hydraulic fluid development section. (Interestingly other structures of PFPAE backbone repeating units required different kettle times to produce the optimum degree of rust.)

## **CREP for Greases**

A convenient test to determine corrosion inhibition characteristics of greases was also needed, as had been developed for liquid lubricants. The test being used was ASTM 1743<sup>7</sup>. ASTM 1743 requires the use of 3 Timkin bearing cone and roller assemblies for each grease to be tested. The bearings are cleaned, packed with grease and then run under a light thrust load for 60 s prior to being stored for 48 hr at 52°C and 100% humidity. The test grease fails if there are one or more spots of rust greater than 1mm in the longest dimension on the bearing cup at the end of the test. The CREP was a very promising test to be adapted for use with greases because it is simpler, costs less, and is quicker than ASTM 1743.

## **Lubricant Application to Test Coupon**

The first aspect of the method to be considered was lubricant application to the metals. Two metals and two greases were used in method development tests. A linear Fomblin Z PFPAE based polytetrafluoroethylene (PTFE) thickened grease was used as the corrosion prone standard and MIL-PRF-32014, a polyalphaolephin (PAO) based lithium soap thickened grease, heavily fortified with rust inhibitors, was used as the corrosion resistant standard. A metal spatula was used to apply grease to all sides of each metal. Next as much of the grease as possible was removed using the metal spatula leaving a thin layer of grease. AISI 1010 metal coupons were evaluated first using water in the reaction kettle. After 1 hour no corrosion appeared on the metals. The test continued to run for 24 hours with no corrosion appearing on either metal. Next after applying and removing grease with a metal spatula, metals were also wiped with a lab wipe to remove more grease from the metal. This process consistently applies approximately 1 to 2 mg of

grease to a metal. As in the previous test no corrosion appeared on either metal in 24 hours.

### **Liquid Test Medium**

To make the corrosive medium more severe the acetic acid/sodium acetate buffer was used in place of distilled/deionized water. Both metals rusted immediately. Pure distilled deionized water was not severe enough but pure acetic acid/sodium acetate buffer (pH=4.63) was too severe so tests were run using different concentrations of buffer in distilled/deionized water until one was found to rust the metal with the corrosion prone standard thoroughly in approximately one hour without rusting the metal with the corrosion resistant standard. A solution of 5% sodium acetate/acetic acid buffer in water was determined to give the optimal discrimination between the corrosion prone and corrosion resistant greases in one hour. (See Table 1.)

### **C-5 Landing Gear Grease**

Corrosion was causing problems in the 300M steel landing gear of C-5 aircraft. Corrosion preventative properties of C-5 landing gear greases needed to be studied and a more corrosion resistant grease found. Metal coupons made of 300M steel were obtained, but because 300M is more corrosion prone than AISI 1010 steel they rusted almost immediately with 5% buffer solution. After trial and error with different buffer concentrations, a 1% solution of buffer in deionized/distilled water was found to discriminate between the corrosion prone and corrosion resistant greases in one hour. To establish these conditions, only two test coupons were used. See Fig. 3.

### **Repeatability and Rater Agreement**

Repeatability for the CREP for greases was tested with 300M steel using 3 coupons. After a working procedure was determined, several trials each of 2 different unknown greases, candidates 1 and 2, were run. Each kettle contained the corrosion prone standard (PFPAE), the corrosion resistant standard (MIL-PRF-32014), and a candidate grease of unknown corrosion inhibiting ability. Two individuals rated these trials independently. See Tables 2 - 3. For the better grease, U1, there was good agreement between the two raters, 8.5 and 8.8, whereas with the poorer grease, U2, there was a larger difference, 5.7 and 3.8. However since both raters found the extent of corrosion to fall well above the value for an acceptable grease, 9.0, the difference is not considered significant.

### **Missile Greases**

M50 and 52100 steels are widely used in missile bearings which are often stored for extended periods of time. A very corrosion protective grease was needed for this application. The CREP buffer/water solution was adjusted to rust standards for each type of metal appropriately. See Table 4. It is much more cost effective to acquire coupons as opposed to bearings for testing. Bearings made from these types of metal would have been necessary to determine the corrosion prevention ability of this grease/metal combination with ASTM 1743.

## **Discussion**

The CREP has been used to screen numerous candidates efficiently, and cost effectively for the following applications: corrosion inhibited engine oils, nonflammable aircraft hydraulic systems development, grease for the C-5 landing gear, perfluoropolyalkylether (PFPAE) oils for high temperature gas turbine oil, and missile greases. Adapting the CREP for these and other applications took some reiterative trial and error experiments but ultimately saved a lot of time for these programs. Using the CREP, except for the 16 hour test time for PFPAE high temperature gas turbine engine oil, duplicate tests could be easily conducted in one day by one person. The CREP for greases has very good repeatability when the good and bad standards rust appropriately. For candidate 1 the average rating difference was less than 1 for the two independent raters and for candidate 2 it was 2 or less, five out of six times. Various types of metal and lubricants have been successfully used with the CREP. Table 5 is a summary of optimized concentrations of buffer solution for various metal types. The CREP is simple, convenient, reliable, and cost effective. Care must be taken in comparing results between different labs. The detailed CREP method is included in Appendix A. For some example results and ratings see Fig. 4.

## Conclusions

1. CREP is a very effective, low cost, time efficient test that can screen many materials quickly.
2. CREP is easy to set up and only requires easily attainable and maintainable equipment that many already have in-house.
3. CREP is useful in comparing many different additives, different concentrations of the same additive, and different base stocks with various metals in a short time frame.
4. CREP is easily adaptable to many applications with different types of lubricants and metals.

## Acknowledgements

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## Appendix Corrosion Rate Evaluation Procedure

### Method

#### 1.0 Purpose

This procedure is used for both liquid and grease lubricants to evaluate corrosion protection. A corrosion resistant standard and a corrosion prone standard are used as references in each test. The oil or grease under test is visually rated against these standards.

#### 2.0 Test specimen preparation

- a. Six metal coupons, measuring 5.08 x 1.27 x 0.16 cm (2 in x 0.5 in x 0.06 in), are required for duplicate tests.
- b. Identify each coupon by imprinting using die-cut 0.32 cm (0.12 inch) numbers or letters.
- c. Clean metal coupons by boiling them in toluene for 5 min, then in boiling acetone for 5 min.
- d. Clean the metal coupons by hand polishing with 240 and then 320 grit silicon carbide papers for 30 s on each side. Briefly sand the edges with 240 grit silicon carbide paper.
- e. Maintain specimen cleanliness by handling with forceps after cleaning.
- f. Final cleaning is accomplished by immersing each coupon in boiling toluene for 5 min., immersing in boiling acetone for five minutes, and then drying in an oven for 30 min.
- g. Store the test coupons in a desiccator for 30 min. to equilibrate to ambient temperature during and after cleaning steps.
- h. Weigh coupons prior to testing.

#### 3.0 Test apparatus assembly

Prepare two identical test set-ups so that duplicate test results may be obtained. See Figs. 1 and 2.

- a. After carefully cleaning and drying all glassware, place the 2 L reaction kettle on the hot plate and add approximately 50 new boiling beads to the reaction kettle to preclude bumping and splashing of the boiling distilled water. (Very clean glassware and new boiling beads are essential as residual oil or additives from previous tests can affect results significantly.)
- b. Place the Teflon® splash suppressor in the reaction kettle so that it is loosely seated against the base of the kettle.
- c. Coat the ground glass flange of the reaction kettle with a thin film of silicone grease to prevent condensate leakage and center the cover on the reaction kettle.
- d. Fit the center female joint with a standard taper 34/45 to 24/40 reducing type bushing adapter.
- e. Insert the male joint of the Allihn condenser into this bushing adapter and insert the male joint of the threaded offset adapter into the female joint at the top of the condenser. Support the condenser as required.

- f. Insert the glass air inlet tube through the center of the Allihn condenser and offset adapter and seal with an o-ring in the threaded bushing of the adapter. Position the air inlet tube so that the flared end is 5.1 cm (2.0 in) above the base of the kettle.
- g. Place the glass stoppers modified with glass hooks in the remaining three female joints of the cover.
- h. Form the coupon suspension hooks, shaped from the AMS 5680 20-gage stainless steel wire, so that the bottom edge of the test coupon is 6 cm from the Teflon separator near the base of the kettle.
- i. Prepare the regulated dry air source using the micrometer valve for flow control and the calibrated flow meter for air flow measurement.

#### 4.0 Test procedure

- a. Start the water flow through the condenser.
- b. Add 100 ml of the appropriate water / buffer (acetic acid/sodium acetate, Fisher Scientific pH 4.63 Certified Buffer) solution to the reaction kettle depending on the type of metal, lubricant and/or specification you are using. The concentration of the buffer solution may vary from 0% (pure deionized/distilled water) to 100% (pure buffer) based on the application as stated above. See Table 5 for suggested concentrations for use with greases with different types of steel. Concentration of buffer may be adjusted as needed to attain the appropriate rusting in one or two hours. For ester based turbine engine oils use 100% buffer. For CTFE and PFAPE fluids use deionized water.
- c. Establish an airflow rate of 500 ml/min to the reaction kettle before the water starts to boil.
- d. Adjust the hot plate to a setting that will result in the reflux occurring halfway up the metal coupon with the air flowing.
- e. Replace one of the glass stoppers with a thermometer in a 24/40 stopper to determine when the temperature has equilibrated.
- f. Allow the system to equilibrate for 60 min. before test specimens are introduced.
- g. Maintain test temperature of the kettle by loosely wrapping the kettle and glassware below the condenser with aluminum foil to avoid cooling by drafts.

#### 4.1 Liquid Application to Coupons

- a. Immerse two of the previously prepared test coupons in each of the three fluids (a test fluid, a corrosion prone standard, and a corrosion resistant standard) for 5 min.
- b. Remove the coupons and suspend in a vertical position in a dust free environment for 15 min.
- c. Remove the excess oil at the bottom edge of the test coupons by light quick dabbing with a wiper.

#### 4.2 Grease Application to Coupons

- a. Apply a thin layer of the unknown grease to the entire surface of two metals with a spatula. Repeat with remaining two greases, the corrosion prone standard and the corrosion resistant standard.
- b. Remove as much grease as possible with a metal spatula.

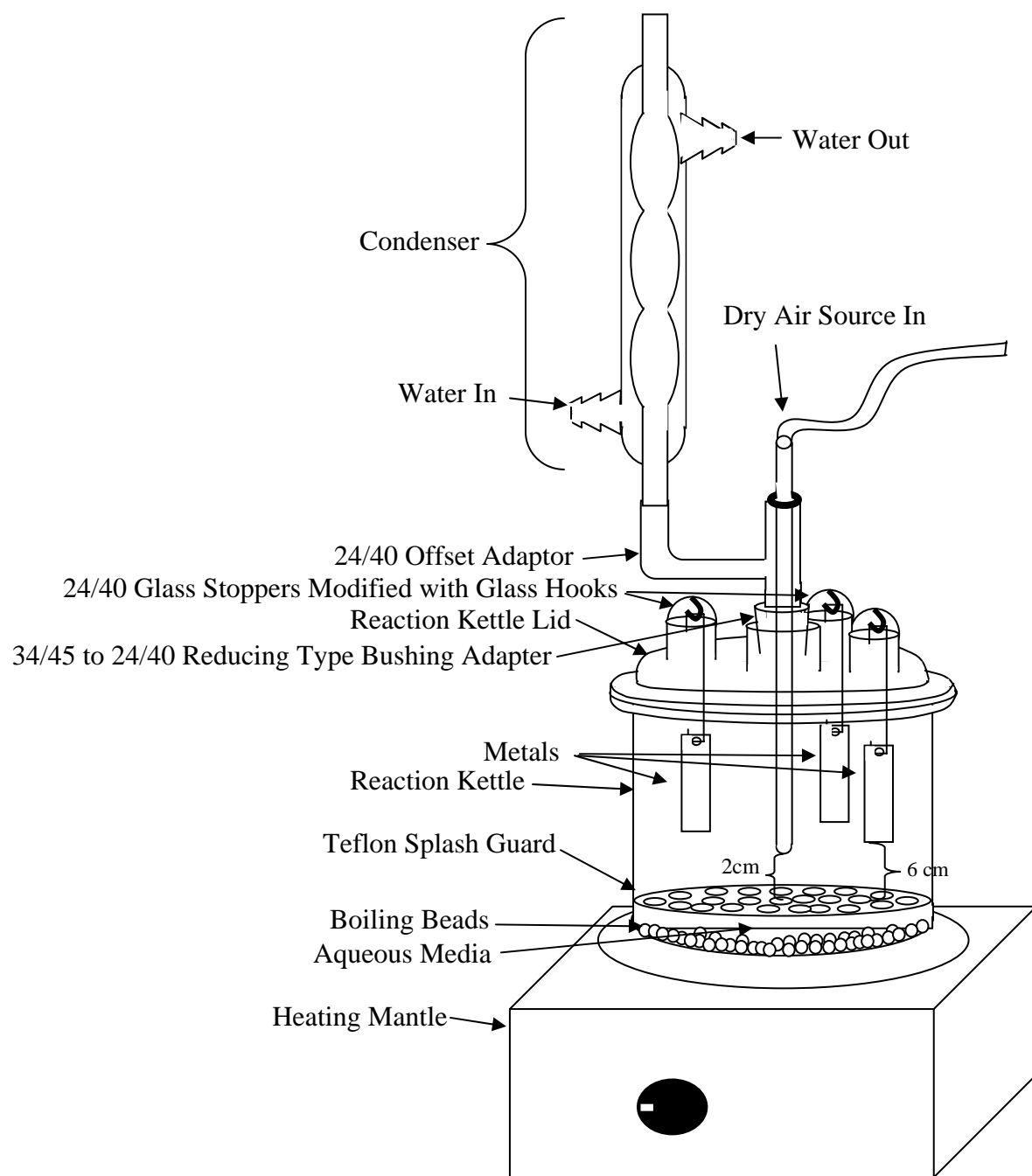
- c. Wipe off as much grease as possible with a lab wiper. This method should apply approximately 1-2 mg of grease to each metal.

#### 4.3 Procedure continued

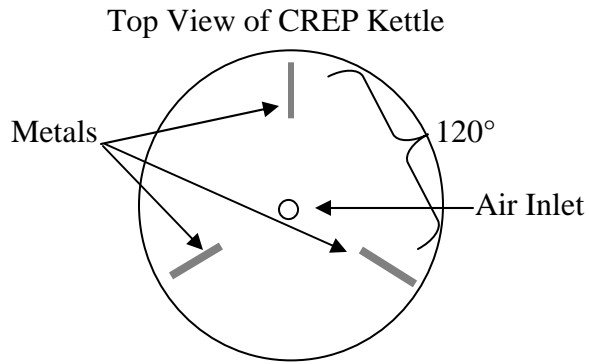
- a. Suspend one set of test coupons (one set coated with each fluid/grease in each reaction kettle) by the wire hooks from the glass stoppers and insert in each reaction kettle. Position the coupons radially from the center of the kettle, 120° apart. See Fig 2.
- b. Remove the test coupons from the reaction kettle after 60 min. Length of time in kettle can be adjusted if necessary for given applications. Photograph each side of the test coupons.
- c. Remove all loose material from the coupons by wiping with a paper tissue or laboratory wipe. Weigh coupon.
- d. Numerically rate the appearance of the coupon of the tested lubricant, comparing it to the coupons that were coated in the corrosion prone and corrosion resistant standards. The coupons corresponding to corrosion resistant standard and corrosion prone standard are rated 10 and 1 respectively. Visual ratings are based on a linear interpolation of the sample coupon compared to the two reference coupons.

#### 5.0 Results

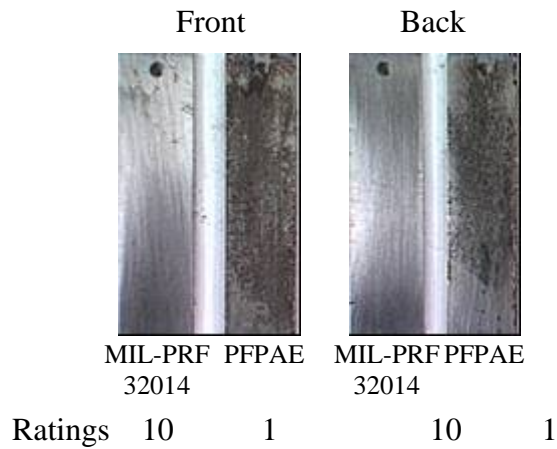
A visual rating of less than 8 is considered a failure. If the two reference standards are not essentially uncorroded for the corrosion resistant standard and severely corroded for the corrosion prone standard, the test results are considered invalid. Repeat the test. Modify the buffer concentration and/or the test time if necessary. A rating difference of more than 2 for duplicate tests is cause for running extra trials.



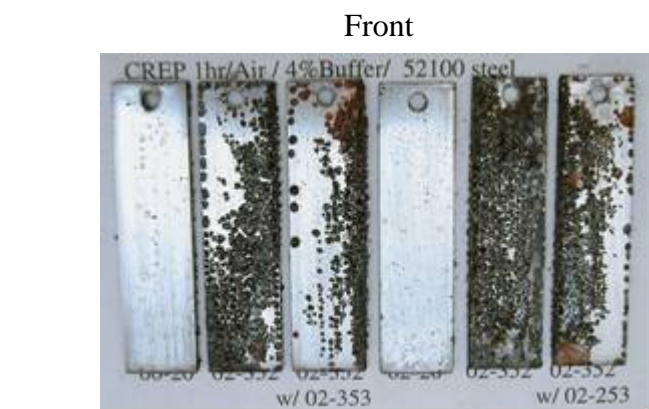
**FIGURE 1.** CREP Experimental Setup (figure not drawn to scale)



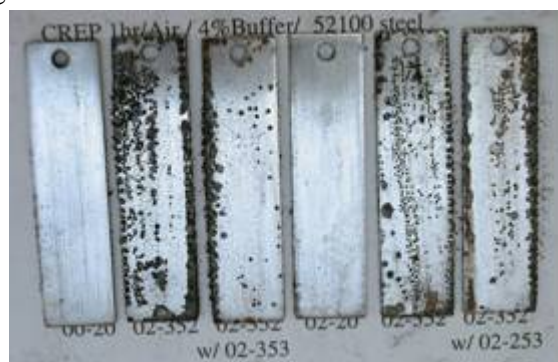
**FIGURE 2.** Alignment of Metals in Kettle



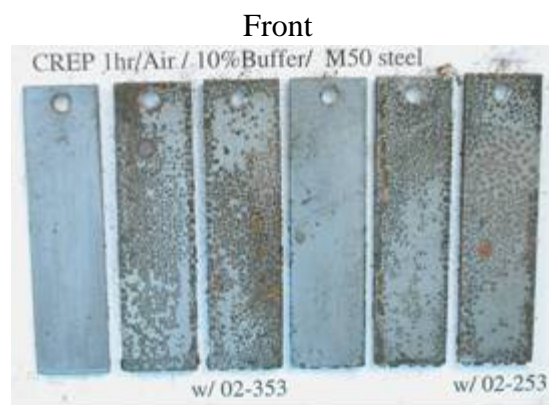
**FIGURE 3.** Photo showing good discrimination between a corrosion prone standard (PFPAGE grease) and a corrosion resistant standard (MIL-PRF-32014) using 300M steel with 1% buffer solution for 1 hour.



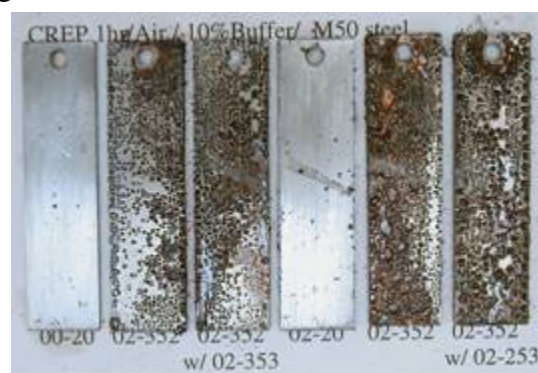
Rating: 10    0    3    10    0    4



Back



Rating: 10    0    0    10    0    0



Back

Test conditions: 1 hour, 500 ml/min airflow  
 Left: 4% sodium acetate buffer, 52100 steel  
 Right: 10% sodium acetate buffer, M50 steel

**Figure 4.** These photos and ratings are examples of CREP results.

<i>% Buffer</i>	<i>Time for Corrosion</i>
50	< 2 minutes
25	< 5 minutes
10	≈1/2 hour
<b>5</b>	<b>≈1 hour</b>
4	≈1 1/2 hours
3	≈2 hours
2	≈3 hours

**TABLE 1.** Optimizing Buffer Concentration  
for Grease Procedure with ANSI 1010  
Steel and PFPAE grease.

Test Date:	11/29	11/29	11/29	11/29	11/29	11/29	11/30	11/30	11/30	11/30	11/30	11/30	Average Rating
Metal ID:	A	B	U1*	A	B	U1*	A	B	U1*	A	B	U1*	Candidate 1
Rater 1:	10	1	10	10	1	9	10	1	8	10	1	8	8.8
Rater 2:	10	1	10	10	1	10	10	1	7	10	1	7	8.5

\* Denotes the unknown grease.

The high corrosion resistance standard A is a MIL-PRF-32014 grease.

The low corrosion resistance standard B is a PFPAE grease.

**TABLE 2.** Grease Candidate 1 Rated by 2 People

Test Date:																		Average Rating	
Metal ID:																		Candidate 2	
Rater 1:	10	1	5	10	1	6	10	1	6	10	1	6	10	1	4	10	1	7	5.7
Rater 2:	10	1	2	10	1	5	10	1	4	10	1	5	10	1	2	10	1	5	3.8

\* Denotes the unknown grease.

The high corrosion resistance standard A is a MIL-PRF-32014 grease.

The low corrosion resistance standard B is a PFPAE grease.

**TABLE 3.** Grease Candidate 2 Rated by 2 People



Conditions	Steel Type	Grease	Rating	Comments	Overall Results
3% Buffer, 1.5 Hr	52100	a	0	Some rust	Not enough Rust
		b	10	No rust	
4% Buffer, 1 Hr	52100	a	0	Coated with Rust	Good Results
		b	10	No Rust	
3% Buffer, 1.5 Hr	M50	a	0	Some rust	Not enough Rust
		b	10	No rust	
5% Buffer, 1 Hr	M50	a	0	Some Rust	Not enough Rust
		b	10	No Rust	
5% Buffer, 1 Hr	M50	a	0	Some Rust	Not enough Rust
		b	10	No Rust	
6% Buffer, 1 Hr	M50	a	0	Some Rust	Not enough Rust
		b	10	No Rust	
10% Buffer, 1 Hr	M50	a	0	Coated with Rust	Good Results
		b	10	No Rust	

a = PFPPE

b = MIL-PRF-32014

**TABLE 4.** Optimizing Buffer Concentration for 52100 and M50 Steel with Grease

Metal	Percent Acetic Acid/Sodium Acetate Buffer in deionized/distilled water	Time
1010	5%	1 hour
52100	4%	1 hour
M50	10%	1 hour
300M	1%	1 hour

**TABLE 5.** Optimal Buffer Solutions Determined with Several Types of Steel for Grease